AFRL-RX-WP-TP-2012-0230

WEAVE AND FIBER VOLUME EFFECTS IN A PIP CMC MATERIAL SYSTEM (Preprint)

G. Ojard, E. Prevost, and R. Naik

Pratt & Whitney

U. Santhosh

Structural Analytics, Inc.

D. C. Jarmon

United Technologies Research Center

FEBRUARY 2012

Approved for public release; distribution unlimited.

See additional restrictions described on inside pages

STINFO COPY

AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
February 2012	Journal Article Preprint	01 November 2011 – 01 November 2011

4. TITLE AND SUBTITLE
WEAVE AND FIBER VOLUME EFFECTS IN A PIP CMC MATERIAL SYSTEM (Preprint)

5a. CONTRACT NUMBER FA8650-05-D-5806-0021

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER 62102F

6. AUTHOR(S)
G. Ojard, E. Prevost, and R. Naik (Pratt & Whitney)
U. Santhosh (Structural Analystics, Inc.)
D. C. Jarmon (United Technologies Research Center)

5d. PROJECT NUMBER 4347

5e. TASK NUMBER 50

5f. WORK UNIT NUMBER LN101100

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Pratt & Whitney 400 Main Street East Hartford, CT 06108	Structural Analystics, Inc. ------------------------------------ United Technologies Research Center

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Research Laboratory
Materials and Manufacturing Directorate
Wright-Patterson Air Force Base, OH 45433-7750
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL/RXLN

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-RX-WP-TP-2012-0230

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
PAO case number 88ABW-2011-6229, cleared 30 November 2011. This work was funded in whole or in part by Department of the Air Force contract FA8650-05-D-5806-0021. The U.S. Government has for itself and others acting on its behalf an unlimited, paid-up, nonexclusive, irrevocable worldwide license to use, modify, reproduce, release, perform, display, or disclose the work by or on behalf of the U. S. Government. Submitted to the Ceramic Engineering & Science Proceedings. Document contains color.

14. ABSTRACT
With the increasing interest in ceramic matrix composites for a wide range of applications, fundamental research is needed in the area of multiple weaves and fiber volume. Understanding how the material performs with differing weaves and fiber volume will affect the end insertion application. With this in mind, a series of three panels were fabricated via a polymer infiltration process: 8 harness satin (HS) balanced symmetric layup, 8 HS bias weave, and angle interlock. From these panels, a series of characterization efforts were undertaken with the sample oriented in both the warp and fill direction: with differing fiber volumes. These tests consisted of tensile, interlaminar shear, interlaminar tensile, in-plane thermal expansion, and through thickness thermal conductivity. In addition, micro-structural characterization was done. The results from this testing will be presented, trends reviewed, and analysis done.

15. SUBJECT TERMS
ceramic matrix composite, fiber architecture, bias weave, angle interlock weave, characterization

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT:	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor)
Unclassified	Unclassified	Unclassified	SAR	18	Paul Jero **19b. TELEPHONE NUMBER** *(Include Area Code)* N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

WEAVE AND FIBER VOLUME EFFECTS IN A PIP CMC MATERIAL SYSTEM

Ojard, G.[1], Prevost, E.[1], Santhosh, U.[2], Naik, R.[1], and Jarmon, D. C.[3]

[1] Pratt & Whitney, East Hartford, CT
[2] Structural Analystics, Inc., San Diego, CA
[3] United Technologies Research Center, East Hartford, CT

ABSTRACT

With the increasing interest in ceramic matrix composites for a wide range of applications, fundamental research is needed in the area of multiple weaves and fiber volume. Understanding how the material performs with differing weaves and fiber volume will affect the end insertion application. With this in mind, a series of three panels were fabricated via a polymer infiltration process: 8 harness satin (HS) balanced symmetric layup, 8 HS bias weave, and angle interlock. From these panels, a series of characterization efforts were undertaken with the sample oriented in both the warp and fill direction: with differing fiber volumes. These tests consisted of tensile, interlaminar shear, interlaminar tensile, in-plane thermal expansion and through thickness thermal conductivity. In addition, micro-structural characterization was done. The results from this testing will be presented, trends reviewed and analysis done.

INTRODUCTION

Ceramic Matrix Composites (CMCs) are being considered for an ever wider range of applications where designers and applications can take advantage of their low density and high temperature capability [1,2]. With this increasing interest, the characterization needs to expand beyond the point design approach used for some potential aerospace applications [3-5]. Based on this, a characterization effort was undertaken to look at a variety of weaves and fiber volumes.

For this effort, three panel types were made available for testing: cross ply balanced panel, bias panel with a ratio of 3:1 and an angle interlock panel. Depending on the orientation of the panel during the testing, the fiber volume varied. These series of panels offered an unique opportunity to perform a consistent set of testing in both orientations and compare weaves and fiber volumes. The following is a report on the testing and characterization that was done.

PROCEDURE

Material

With the interest in exploring multiple weaves and fiber volumes, a polymer infiltration pyrolysis system was chosen. This was due to the ability of the process to be easily transferred to the different weaves with no modification to the processing or processing time eliminating that as a variable in any subsequent analysis. The material system used for this effort was the SiC/SiNC system which a non-stochiometric SiC (CG Nicalon™) fiber in a matrix of Si, N and C that is arrived at by multiple iterations via a polymer pyrolysis process. This material has been previously discussed by the authors where the constituent properties were determined [6].

The baseline panel for this effort was a cross ply panel using a 22 ends per inch (epi) 8 HS balanced cloth. The panel was a 6 ply panel with an overall fiber volume set at 40%. The second panel was a Bias weave panel (cross ply layup at 6 plys) where the warp fibers were set

at ~3x the fill direction fibers. The overall fiber volume was set at 40% (consistent with the baseline panel). The Angle Interlock panel was an effort to combine a bias weave with a low angle interlock to increase interlaminar properties. The total fiber volume was set at 35%. The manufacturing goals for the fiber volume which shows the nature of the various weaves is found in Table I.

Table I. Manufacturing Goals for Fiber Volume between the Three Panels

Panel Type	Fiber Direction	Fiber Volume (Vf)
		From Manufacture
		(%)
Baseline	Warp	20.1
	Fill	20.1
Bias	Warp	28.8
	Fill	11.1
Angle Interlock	Warp	24.5
	Fill	10.5

Test Matrix

For each weave, the overall panel size was 310 mm x 310 mm. This provided a large enough area that samples could be machined in both the warp and fill direction to not only look at the weave but also fiber volume: warp and fill. For tensile testing, there were 6 samples from each direction with test temperatures of 24°C, 649°C and 982 °C. This resulted in two repeats for each condition. There were two interlaminar tests performed. The interlaminar tensile testing was done at 24°C with 4 repeats per panel. There were 6 samples for the interlaminar shear testing for each direction for testing at 649°C and 982°C. This resulted in three repeats for each condition. For the in-plane thermal expansion, there were 3 repeats for each test direction with the testing done from 24°C to 982 °C. There were only 2 through thickness thermal conductivity tests done for each panel type. In all cases, the testing was done per ASTM standards (as shown in Table II).

Table II. Characterization Matrix for Panel (planned for each panel type)

Test Description	Test	Temp	Test	Test	Reps
	Direction	**(°C)**	**Environment**	**Method**	
Tensile	Warp & Fill	24	air	C1275	2
Tensile	Warp & Fill	649	air	C1359	2
Tensile	Warp & Fill	982	air	C1359	2
Interlaminar Shear	Warp & Fill	649	air	D2344	3
Interlaminar Shear	Warp & Fill	982	air	D2344	3
Interlaminar Tensile	Normal	24	air	D7291	4
In Plane Thermal Expansion	Warp & Fill	24-982	inert	E228	2
In Plane Thermal Expansion	Warp & Fill	24-982	inert	E228	2
Through Thickness Conductivity	Normal	24-815	inert	E1225	2

Characterization:

After both fabrication or testing, characterization efforts were undertaken. Standard micro-structural characterization was done samples from the panels looking at the microstructure in the warp and the fill directions. In addition, image analysis was done to determine the fiber volume and percent porosity seen in the images. Scanning electron microscopy was performed on the failed tensile bars to look for differences that would occur due to weave or fiber volume effects.

RESULTS

Tensile Testing

The room temperature tensile results for the three panel types are shown in Figures 1-3. The average data for all the temperatures is shown in Table III. For the baseline panel, the testing does not show any significant difference between the sample directions. This is not the case for the Bias or Angle Interlock panels. These two panel types showed that there was a significant decrease in the strength and the proportional limit due to the decreased fiber volume present in the fill direction (see Table I). The table and curves show that there still was significant strain to failure capability present (as compared to the warp direction for these panels).

Interlaminar Testing

As noted in Table II, there were two interlaminar tests run on the material. The interlaminar shear testing was performed in a 4 point short beam shear test. The test series run did not generate the correct shear failure mode per the ASTM standard and therefore will not be discussed further. There were also a series of 4 interlaminar tensile tests run on each panel. The results of that testing is shown in Table IV. Table IV shows that there were no differences seen between the various panels fabricated.

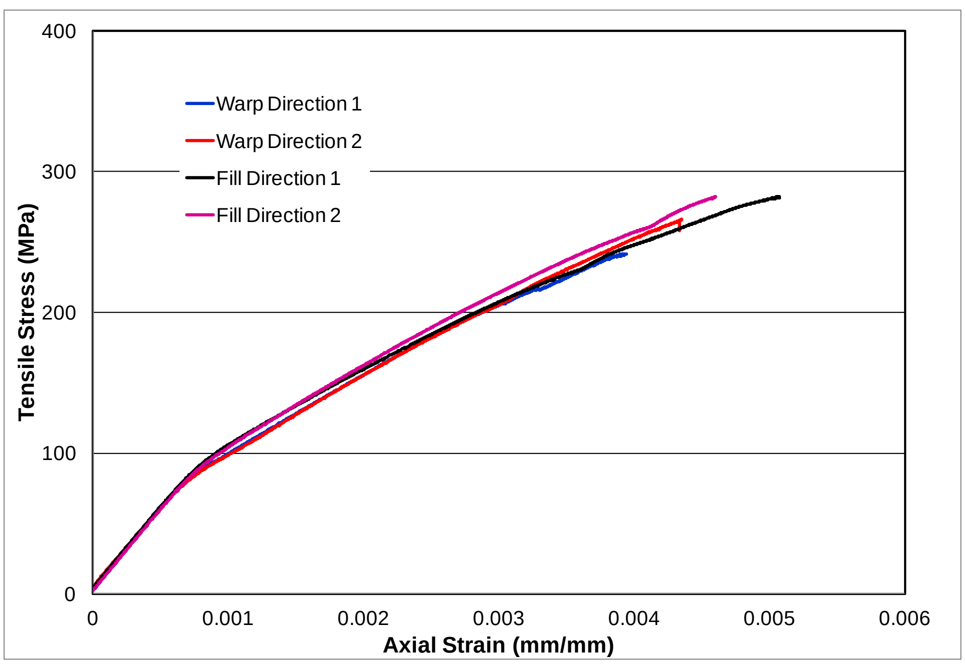


Figure 1. Room Temperature Stress Strain Curved from Baseline 8 HS Cross Ply Panel

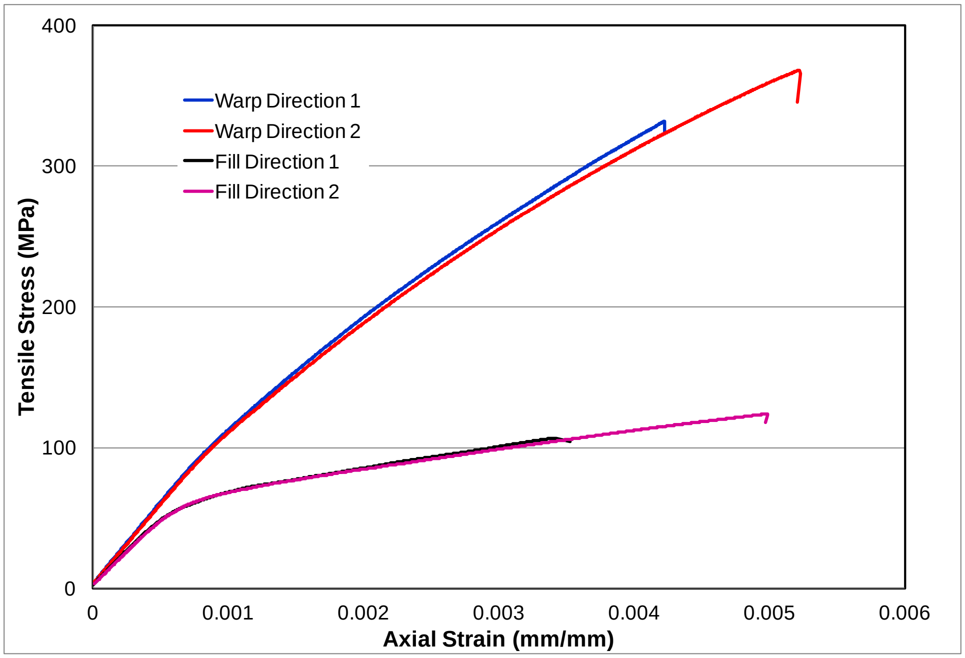


Figure 2. Room Temperature Stress Strain Curved from Bias Panel

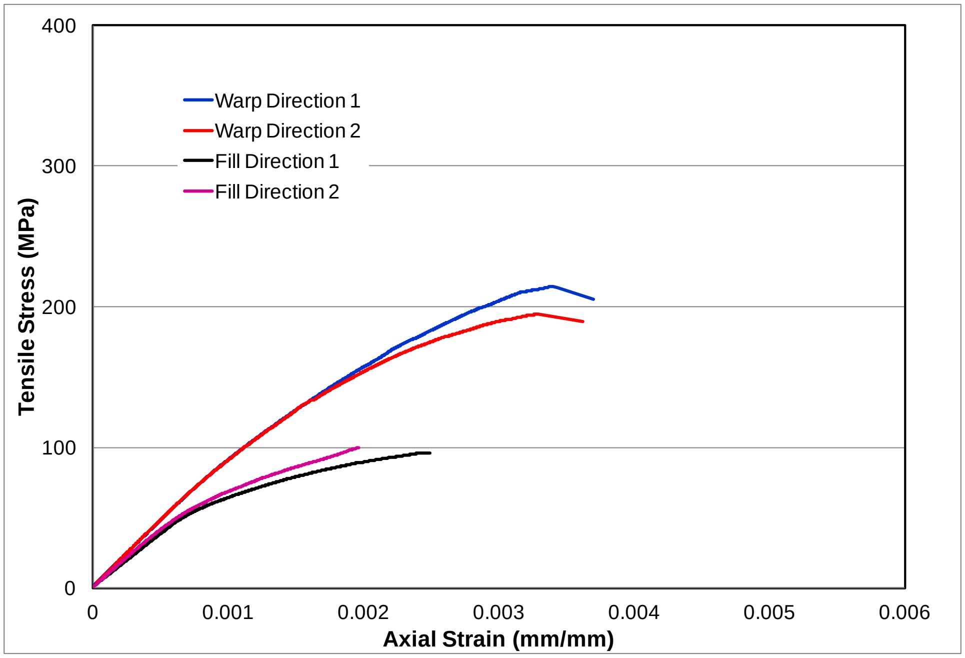


Figure 3. Room Temperature Stress Strain Curved from Angle Interlock Panel

Table III. Average Tensile Data for Three Panel Types

Panel Type	Test Direction	Test Temp	Proportional Limit	UTS	Failure Strain	Modulus
		(°C)	(MPa)	(MPa)	(mm/mm)	(GPa)
Baseline	Warp	24	84.6	253.7	0.0041	114.9
Baseline	Fill	24	92.8	282.3	0.0049	115.9
Baseline	Warp	649	77.3	300.3	0.0052	121.4
Baseline	Fill	649	89.3	298.7	0.0049	125.2
Baseline	Warp	982	82.3	302.3	0.0054	120.1
Baseline	Fill	982	90.6	320.9	0.0063	111.8
Bias	Warp	24	109.0	350.0	0.0047	109.0
Bias	Fill	24	52.1	115.4	0.0042	98.0
Bias	Warp	649	na	na	na	na
Bias	Fill	649	42.8	113.2	0.0047	107.0
Bias	Warp	982	101.0	335.7	0.0047	124.9
Bias	Fill	982	42.2	116.5	0.0055	120.8
Angle Interlock	Warp	24	89.9	204.5	0.0034	95.2
Angle Interlock	Fill	24	53.6	98.0	0.0022	80.4
Angle Interlock	Warp	649	90.4	216.8	0.0039	101.8
Angle Interlock	Fill	649	61.2	108.3	0.0036	66.9
Angle Interlock	Warp	982	88.4	227.8	0.0047	94.9
Angle Interlock	Fill	982	51.9	126.7	0.0054	76.9

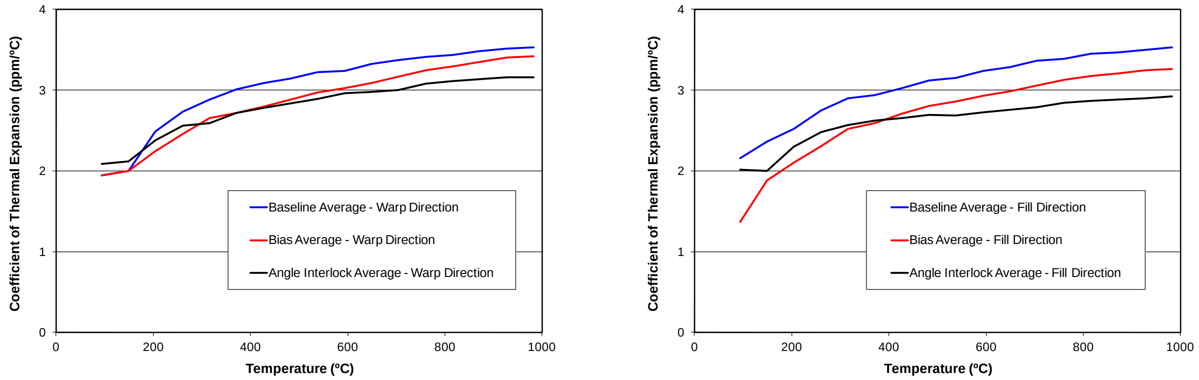
na = delamination present in panel reduced the samples present for testing and therefore this temperature was removed from the test matrix

Table IV. Interlaminar Tensile Average for Three Panel Types (with standard deviation)

Panel	Analysis	ILT Strength (MPa)	Failure Location
Baseline	Average	14.1	100% Material
	StDev	1.08	
Bias	Average	12.9	100% Material
	StDev	3.90	
Angle Interlock	Average	14.9	100% Material
	StDev	0.28	

Thermal Testing

A series of in-plane thermal expansion tests were done in both the warp and fill directions. The results of this testing are shown in Figure 4. There is not a clear distinction between the test directions with the known change in fiber volume. The results of the thermal conductivity testing are shown in Figure 5. Here, clear differences are seen with the angle interlock sample showing the greatest conductivity value.



a) In Warp Direction b) In Fill Direction

Figure 4. In-Plane Thermal Expansion Results for the Three Panel Types

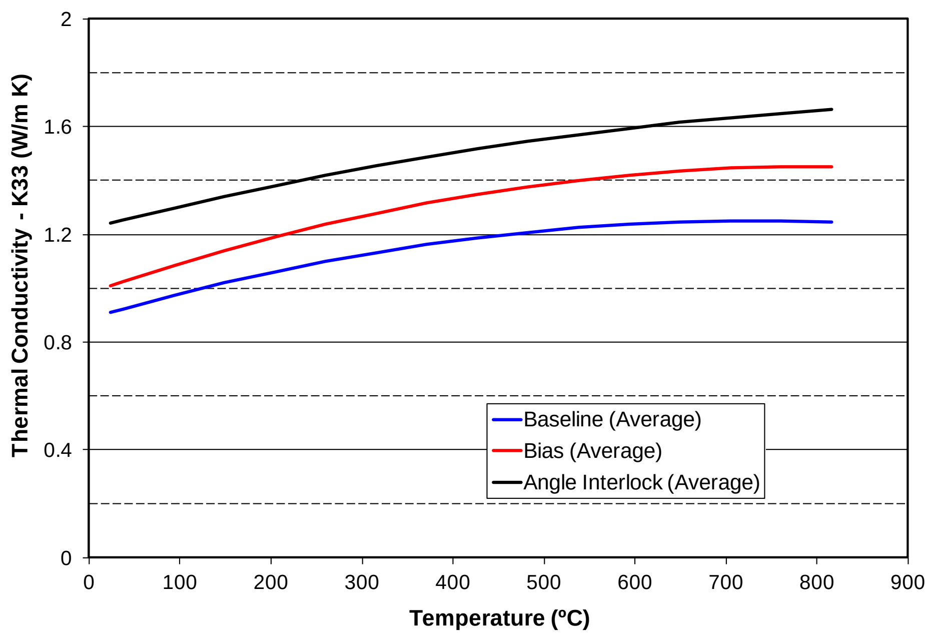
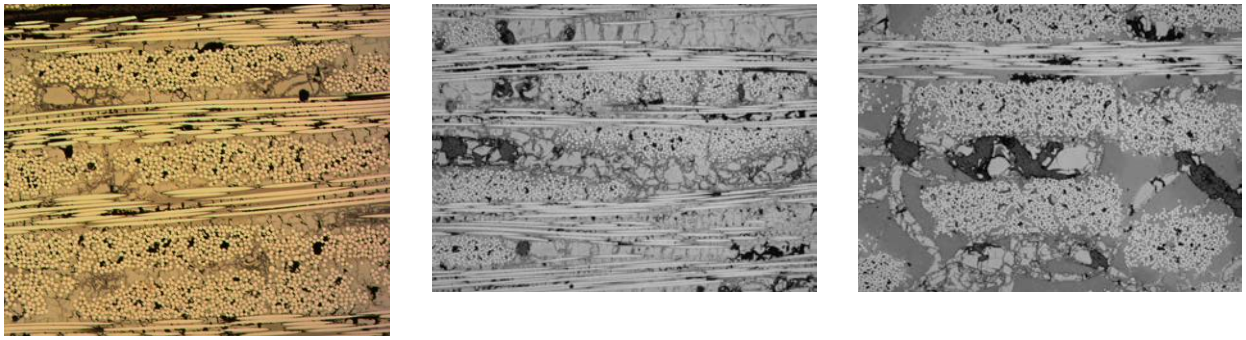


Figure 5. Through Thickness Thermal Conductivity Results

Characterization

As noted previously, micro-structural characterization was performed on the material. From the optical work, image analysis was done to determine fiber volume on the baseline panel and porosity analysis for all the panels. Micro-structural cross sections showing the fill direction for the three panel types are shown in Figure 6. Figure 6 shows that the fill direction has different fiber volumes as expected. From these images, porosity analysis was done and the summary of

that work is shown in Table V. The porosity is essentially constant for this effort and will not be discussed further. For this series of work, only the fiber volume for the baseline panel was determined and it was found to be 24.7% which is higher than expected from the manufacturing expectations shown in Table I.



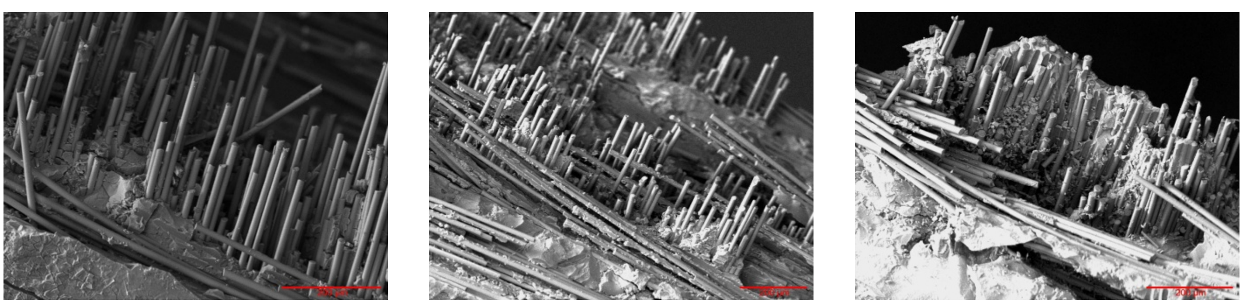
a) Baseline (50x) b) Bias (50x) c) Angle Interlock (50x)

Figure 6. Optical Images of Fill Direction for the Three Panel Types

Table V. Porosity Analysis (Image) for the Three Panel Types

Panel Type	Fiber Direction	Porosity
		(%)
Baseline	Warp	8.4
	Fill	11.6
Bias	Warp	8.2
	Fill	11.1
Angle Interlock	Warp	8.8
	Fill	11.2

After tensile testing, Scanning Electron Microscopy (SEM) was performed on the tensile fracture faces to confirm the presence of fiber pullout. Images from the testing in the fill direction are shown in Figure 7. Fiber pullout is seen consistent with the strain to failures (See Table III.)



a) Baseline b) Bias c) Angle Interlock

Figure 7. SEM Images of Tensile Samples Tested in the Fill Direction
(tested at RT)

Mechanical Characterization

In addition to the optical characterization, the tensile testing was used to determine the fiber volume in the different loading directions. This can be done by looking at the secondary slope of the stress strain curve after the proportional limit. After the proportional limit, all the load is being carried by the fiber and the slope is equivalent to the fiber volume times the modulus of the fiber [7,8]. The results of this analysis are shown in Table VI. There is good agreement with the expectations from manufacturing. The mechanical testing generates higher values in the warp direction and lower values in the fill direction. The testing done for the baseline panel was confirmed using optical image analysis as discussed above.

Table VI. Mechanical Analysis of Fiber Volume for the Three Panel Types

Panel Type	**Fiber Direction**	**Vf**	**Vf**
		Manufacture	**Tensile Curve**
		(%)	(%)
Baseline*	Warp	20.1	24.7
	Fill	20.1	24.7
Bias	Warp	28.8	35.1
	Fill	11.1	6.9
Angle Interlock	Warp	24.5	21.0
	Fill	10.5	8.4

* = optical analysis was done on the baseline panel and the determined fiber Vf was 24.7*

DISCUSSION

Three different panel types were tested in two different directions with some testing done in the through thickness direction of the panels. The breadth of this work allows the data to be compared against fiber volume and weave effects. This is shown in Figures 8-10 where the modulus, proportional limit (0.005% offset) and ultimate tensile strength can be viewed against fiber volume. (For the Elastic Modulus, past work was used for the constituent properties allowing a Rule of Mixtures (ROM) approach to be used [6].) These series of results clearly show that the fiber volume can have a significant impact on the properties. The elastic modulus is consistent with the Rule of Mixtures especially when the porosity in the material was clearly known (See Table V). The change in the fiber volume clearly influences the ultimate tensile strength. With increasing percentage of high strength fibers, there is a clear benefit of the fiber seen. The proportional limit increases with fiber volume and this is most likely due to a decrease in the amount of the weak matrix present.

The interlaminar properties did not clearly differentiate between the panels and samples tested. As noted earlier, the interlaminar shear did not have the correct failure mode so no analysis can be done. The interlaminar tensile testing showed no difference between the panels even with the angle interlock work done (See Table IV). The presence of porosity is not an issue here as the panels were consistent as noted earlier.

While the angle interlock did not improve the mechanical properties in the through thickness direction, there was a benefit seen for the thermal conductivity (See Figure 5). The presence of fibers in the Z (through thickness) direction allowed improved thermal conductivity over the other panels that relied on the matrix for part of the through thickness thermal conductivity. The thermal expansion testing did not show significant changes but this is most likely due to the

expansions between the two phases being the same. This needs to be confirmed with experimental work.

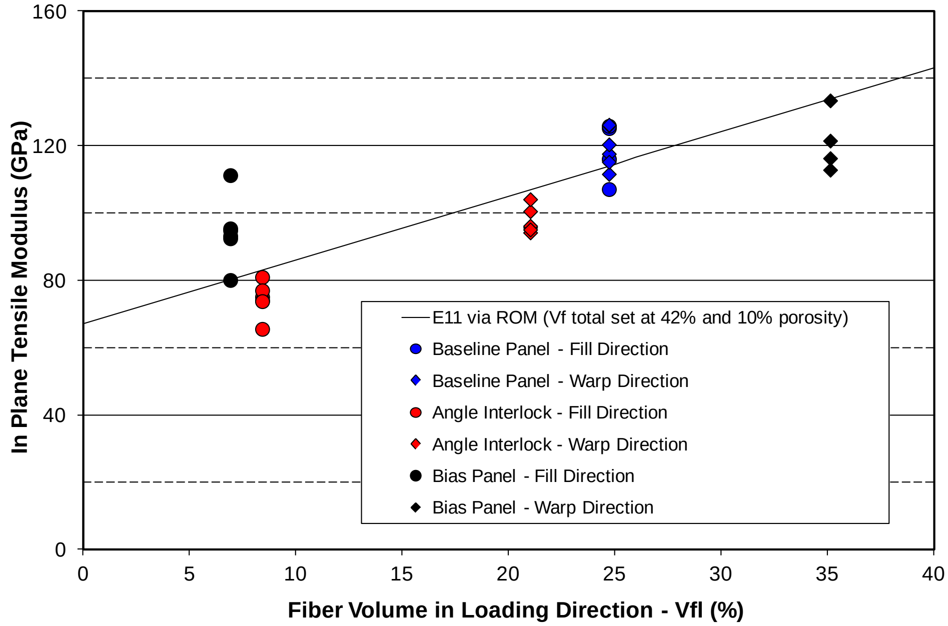


Figure 8. Tensile Modulus versus Fiber Volume
(with ROM Theory Shown)

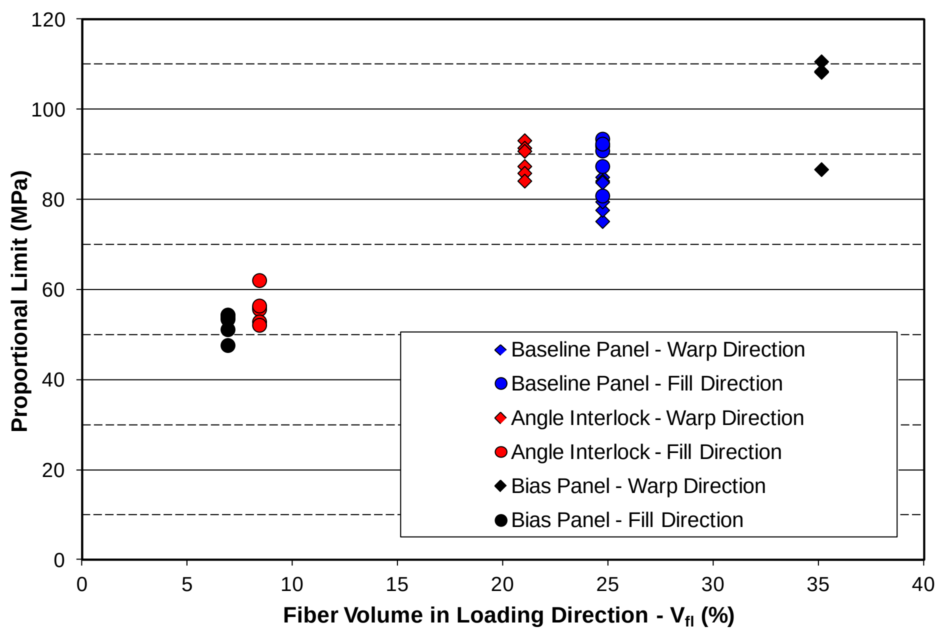


Figure 9. Proportional Limit versus Fiber Volume

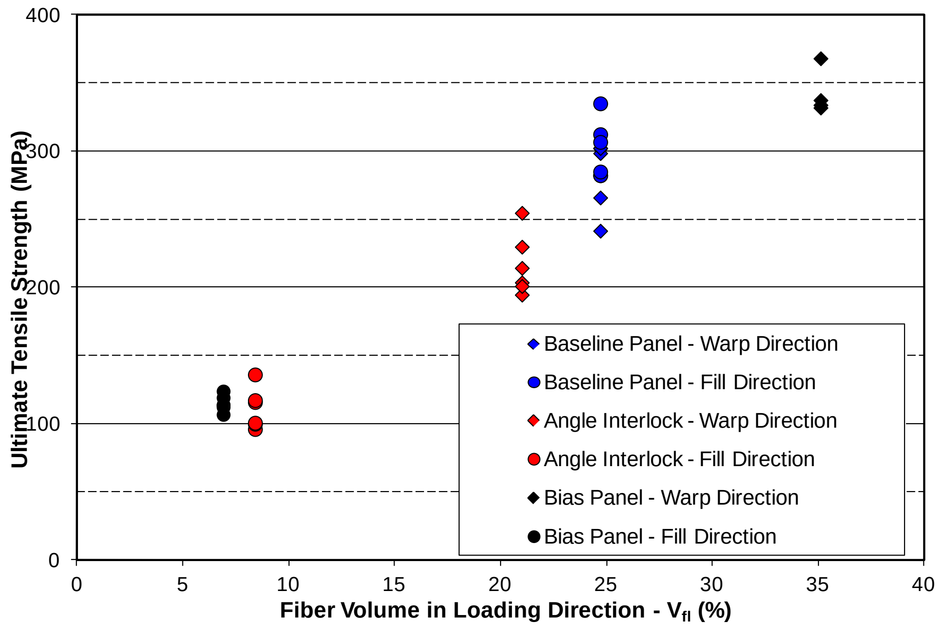


Figure 10. Ultimate Tensile Strength versus Fiber Volume

CONCLUSION

A series of tests were conducted on an 8HS baseline panel, a bias panel and an angle interlock panel. Testing was done in both the warp and fill directions. Fiber volume was determined form the testing and used to look at trends with fiber volume. This testing showed that the mechanical properties were greatly influenced by fiber volume. The interlaminar properties were not affected. There was some effect on thermal properties for the through thickness thermal conductivity for the angle interlock panel due to the presence of Z direction fibers.

This work shows the benefit of looking at a range of weaves and fiber volume as this can impact the design space for future applications.

FUTURE WORK

As part of the effort to understand this data, additional constituent property testing should be done. As noted above, this is clearly the case for the thermal expansion where the data versus temperature is not fully known.

ACKNOWLEDGMENTS

Work performed under the Ceramic Matrix Composite Turbine Blade Demonstration Program, Contract FA8650-05-D-5806 Task 0021, Dr. Paul Jero program manager.

REFERENCES

1. K.K. Chawla (1998), ***Composite Materials: Science and Engineering, 2nd Ed***., Springer, New York
2. Ashby, Michael F. (2005). Materials Selection in Mechanical Design (3rd Edition). (pp: 23 (need to look at book and add in page numbers). Elsevier.
3. Brewer, D., 1999, “HSR/EPM Combustion Materials Development Program”, Materials Science & Engineering, 261(1-2), pp. 284-291.
4. Brewer, D., Ojard, G. and Gibler, M., “Ceramic Matrix Composite Combustor Liner Rig Test:, ASME Turbo Expo 2000, Munich, Germany, May 8-11, 2000, ASME Paper 2000-GT-670
5. Verrilli, M. and Ojard, G., “Evaluation of Post-Exposure Properties of SiC/SiC Combustor Liners testing in the RQL Sector Rig”, Ceramic Engineering and Science Proceedings, Volume 23, Issue 3, 2002, p. 551-562.
6. Ojard. G., Rugg, K., Riester, L., Gowayed, Y. and Colby, M., “Constituent Properties Determination and Model Verification for a Ceramic Matrix Composite Systems”, Ceramic Engineering and Science Proceedings. Vol. 26, no. 2, pp. 343-350. 2005.
7. Ojard, G., Gowayed, Y., Morscher, G., Santhosh, U., Ahmad, J., Miller, R. and John, R., “Frequency And Hold-Time Effects On Durability Of Melt-Infiltrated SiC/SiC”, to be submitted to Ceramic Engineering and Science Proceedings, 2011.
8. Courtney (per textbook)